Yield Symmetry and Reduced Strength Differential in Mg-2.5Y Alloy

Dalong Zhang^a, Haiming Wen^{b, c}, M. Arul Kumar^d, Fei Chen^e, Lianmeng Zhang^e, Irene J. Beyerlein^f,
Julie M. Schoenung^a, S. Mahajan^g, and Enrique J. Lavernia^{a*}

^aDepartment of Chemical Engineering and Materials Science, University of California-Irvine,
Irvine, CA, 92697-2575, USA

^bDepartment of Physics, Nuclear and Electrical Engineering, Idaho State University, Idaho Falls,
ID 83402, USA

^cCharacterization and Advanced PIE Division, Idaho National Laboratory, Idaho Falls, ID 83415,
USA

^dMaterials Science and Technology Division, Los Alamos National Laboratory, Los Alamos,
NM 87545, USA

^eState Key Lab of Advanced Technology for Materials Synthesis and Processing, Wuhan
University of Technology, Wuhan 430070, People's Republic of China

^fTheoretical Division, Los Alamos National Laboratory, Los Alamos, NM 87545, USA

^gDepartment of Chemical Engineering and Materials Science, University of California-Davis,
Davis, CA 95616, USA

*Corresponding author. E-mail address: lavernia@uci.edu (E.J. Lavernia)

Abstract

In this study we report novel results obtained with an extruded fine-grained Mg-2.5 at.% Y alloy (FG Mg-2.5Y) exhibiting tension/compression yield symmetry and reduced strength differential, in addition to well-balanced strength and ductility. On the basis of detailed post-mortem transmission electron microscopy studies, atom probe tomography, and electron back-scattered diffraction (EBSD) characterization, we propose that the presence of a supersaturated solid solution strengthening for basal slip, and the enhanced activity of prismatic slip are the major causes for the unusual mechanical behavior.

Keywords: magnesium alloy; texture, dislocation imaging; tension/compression asymmetry; crystal plasticity

1. Introduction

Unlike cubic metallic materials, hexagonal close packed (HCP) Mg alloys often exhibit significantly different plastic flow characteristics under tension and compression [1-3]. For example, deformed along the extrusion direction (ED), typical Mg alloys exhibit a compressive yield strength (CYS) that is lower than its tensile yield strength (TYS). However, more significant work-hardening occurs in compression, resulting in higher ultimate compressive strength (UCS) than ultimate tensile strength (UTS) [4]. The former aspect of this tension/compression asymmetry is often called a yield asymmetry, which is characterized by the ratio of CYS and TYS; whereas the latter is called (tension/compression) strength differential. The physical explanation for yield asymmetry is that in an extruded Mg alloy tested along the ED, which usually contains a basal texture [4], the dominant deformation modes in tension are non-basal slip plus basal slip, while in compression it is twinning plus basal slip [4, 5], and the twinning stress is usually lower than the stress to activate non-basal slip [6, 7].

The yield asymmetry and related tension/compression strength differential [8] phenomena due to basal texture largely limit the application of Mg alloys [9]. Therefore, efforts have been devoted to address these issues [9-14]. A survey of the published literature reveals that currently there are two major approaches. The first one is randomizing the texture, namely making the bulk materials more isotropic by one or more of the following methods: tailoring the texture by alternative processing routes and/or heat treatments, such as enhancing recrystallization [9], equal-channel angular extrusion [11], twin-roll casting [12], etc.; or alloying Mg or Mg alloys with rare earth (RE) elements, such as Ce, La, Y [6, 10, 15], to weaken the basal texture. In this way, the bulk polycrystalline Mg alloys would be more isotropic, which leads to less pronounced yield asymmetry due to similar deformation modes under both tension and compression. The second approach involves introducing second phases to basal-textured Mg alloys, such as the quasi-crystal phases in $\text{Mg}_{93}\text{Zn}_6\text{Y}$, $\text{Mg}_{93}\text{Zn}_6\text{Ho}$ and $\text{Mg}_{96.9}\text{Zn}_{2.7}\text{Ho}_{0.4}$ alloys [13, 14], or the $\text{Mg}_{17}\text{Al}_{12}$ precipitates in Mg-8Al-0.5Zn and AZ91 alloys [16]. In this case, even though the often undesirable basal texture may still exist, the second phases are able to impede both non-basal slip and twinning, thus improving yield strength (YS), especially CYS, and thereby reducing yield asymmetry. It is noted that alloying elements (whether RE elements or not) may also cause random texture besides forming precipitates [9, 17]. While the mechanisms associated with the

first approach are well understood, the interaction between the second phases/precipitates and non-basal slip [13, 14, 16, 18] and/or twinning[13, 14, 19] remains poorly understood. Nevertheless, the above-mentioned approaches could at best reduce the yield asymmetry only, while the overall tension/compression strength differential usually remains significant in wrought Mg alloys. For instance, in a study on twelve different extruded Mg alloys[10], it was reported that while the values of CYS/TYS were near unity, the fracture strains and ultimate strengths in tension and compression were remarkably different.

In this study, we propose a novel approach to effectively reduce tension/compression asymmetry in the overall deformation response, rather than just on yield strength. This entails alloying Mg with Y via powder metallurgy processes[20, 21], followed by conventional extrusion. It is worth noting that the presence of Y, not only reduces the basal plane stacking fault energy (SFE) in Mg thus enhancing the population of basal stacking faults (BSFs)[20, 22], but also reduces basal texture development in wrought Mg alloys [23], and promotes non-basal slip [24], as well as significant solid solution strengthening [25]. The modified texture and well balanced slip activities result in virtual yield symmetry and reduced strength differential.

2. Experimental Procedure

Fine-grained Mg-2.5 at.% Y alloy (FG Mg-2.5Y, hereafter) was prepared via powder metallurgy[26, 27] followed by conventional extrusion at 350 °C. It is worth noting that the equilibrium solubility of Y in Mg at ambient temperature is ~0.6 at.%[28], which is below the solubility limit of ~2.5 at.% at 350 °C [29]. However, by employing non-equilibrium thermo-mechanical processing, including gas atomization and hot extrusion [30], an Mg-Y alloy with supersaturated Y atoms in solid solution may be attained and hence the unique effects of Y on Mg matrix could be fully leveraged. Cylindrical tensile test samples with a gauge length of 10 mm and diameter of 3 mm were tested along the extrusion direction (ED), whereas cylindrical compression test samples, with 5 mm in diameter and 7 mm in height, were also tested along the ED.

The microstructure of the as-extruded FG Mg-2.5Y (AE sample hereafter) was characterized using electron backscattered diffraction (EBSD, Oxford Instruments EBSD detector located in a FEI Scios scanning electron microscope) and a JEOL 2500 transmission electron microscope

(TEM).EBSD specimens were prepared by a JEOL cross-section polisher (Model SM-09010), whereas TEM specimens were sectioned, mechanically ground, and thinned in an ion mill (Gatan PIPS).For the AE sample, the TEM thin foil specimens were sectioned either normal to the ED or parallel to the ED, in order to observe grains of different orientations. Atom probe tomography (APT) was also performed on the AE sample, using a Cameca local-electrode atom-probe (LEAP) 4000X-Si tomograph. Electropolishing was used to obtain taper-shaped sharp tips with a radius of curvature of ~50 nm at the tip apex. During APT data collection, field evaporation was assisted by an ultraviolet picosecond pulsed laser that has a wavelength of 355 nm, a pulse repetition rate of 200 kHz and a pulse energy of 5-8 pJ. Steady-state DC voltages between 2.0 and 6.0 kV were applied, and the specimen base temperature was 30 K. A straight flight path system was utilized for APT measurements, and the ion flight distance was 90.0 mm. The evaporation rate was 0.5-2.5%. Cameca's IVAS[®] software, version 3.6.4, was utilized to reconstruct and analyze the APT data. More details regarding APT can be found in Refs.[31, 32].

EBSD was also employed to track the evolution of deformation texture, including samples compression-tested to ~2% true strain (denoted as the Comp-2% sample hereafter), and tensile-tested to ~2% true strain (denoted as the Tens-2% sample hereafter). All EBSD specimens were sectioned in such a way that the viewing planes were parallel to the ED, and polished by a JEOL SM-09010 cross section polisher with Ar ion beam. Due to the small grain size and the various amount of strain in EBSD specimens, the indexing rate varied between 65% and 75%. The EBSD data were analyzed with Oxford HKL software. In addition, detailed TEM studies were performed for the Comp-2% sample as well, and the TEM thin foil specimen was sectioned normal to the ED.

For the TEM study, two-beam bright-field (TBBF), two-beam dark-field (TBDF) or weak-beam dark-field (WBDF) imaging techniques were used to obtain dislocation contrast[33]. In addition, the “ $\mathbf{g} \cdot \mathbf{b}$ ” visibility criteria was used to discern different types of dislocations in HCP lattice, i.e., $\langle a \rangle$ type with $\mathbf{b} = \frac{1}{3} \langle 11\bar{2}0 \rangle$, $\langle c \rangle$ type with $\mathbf{b} = [0001]$, and $\langle c+a \rangle$ type with $\mathbf{b} = \frac{1}{3} \langle 11\bar{2}3 \rangle$ [34-36]. Particularly, the $\mathbf{g} = 0002$ condition is especially useful, as $\langle a \rangle$ type dislocations are invisible (with $\mathbf{g} \cdot \mathbf{b} = 0$), whereas only dislocations having a $\langle c \rangle$ component (i.e., $\langle c \rangle$ or $\langle c+a \rangle$ type) will be in contrast[35].

3. Results

3.1 Texture, microstructure and chemical composition in as-extruded FG Mg-2.5Y

Figure 1 shows the grain size and texture of the AE sample through a colored EBSD map. The color code (Fig. 1(a)) is based on the angle (ϕ hereafter, ranging $0^\circ \sim 90^\circ$) between the c-axis of each grain and the ED (i.e., the horizontal direction herein). Namely, the red grains have c-axis approximately normal to the ED (i.e., ϕ between $75^\circ \sim 90^\circ$), and blue grains have c-axis approximately parallel to the ED (i.e., ϕ between $0^\circ \sim 15^\circ$), whereas the green/yellow/orange grains have ϕ values in between (i.e., ϕ between $15^\circ \sim 75^\circ$). It is noted that the average grain size is $\sim 1 \mu\text{m}$ based on statistics over 15,000 grains. Therefore, our powder metallurgy processing resulted in much finer grain size than that of Mg-RE alloys produced by conventional casting and rolling/extrusion [22, 37].

In addition, Fig. 1(b) is the inverse pole figure (IPF) based on the map data in Fig. 1(a). On the one hand, there exists a weak extrusion basal texture (the red area) [38] with the maximum MUD (multiple of uniform distribution) of 2.68; on the other hand, there seems to be another texture component (the green area) similar to the well-known “rare-earth” texture [23, 38]. To better quantify and visualize the texture distribution, the percentage of each ϕ values (grouped by interval of 5°) is plotted in Fig. 1(c). It is clear that the $75^\circ \sim 80^\circ$, $80^\circ \sim 85^\circ$, and $85^\circ \sim 90^\circ$ intervals account for the highest percentages: $\sim 11\%$, $\sim 12\%$, and $\sim 13\%$, respectively. Since basal texture can be defined as ϕ values within $75^\circ \sim 90^\circ$ [39], the basal texture fraction is then estimated to be $\sim 36\%$. However, there is a large fraction of grains ($\sim 50\%$ in total) having ϕ values between $45^\circ \sim 75^\circ$. That is, about half of the grains have their c-axis inclined relative to the ED. Therefore, this texture component could be regarded as the primary texture component, whereas the basal texture component as the secondary texture component. In fact, the average ϕ value over the large dataset in Figure 1 is calculated to be $\sim 65^\circ$. Overall, it is noted that the AE sample was mostly recrystallized with “rare-earth”-like primary texture component represented by fine green/yellow grains, whereas the weak extrusion basal texture (i.e., secondary texture component) was maintained and represented by the fine red grains as well as the few large red grains [38].

The detailed microstructure of many grains with various orientations was studied in detail by TEM, as shown in Figure 2 and Figure 3. Figure 2 presents one grain (Grain X1 hereafter) near the $[2\bar{1}\bar{1}0]$ zone axis with minimum tilting, as shown in Fig. 2(a) and inserted diffraction pattern. As this TEM specimen foil was sectioned parallel to the extrusion direction (ED) and the ED is

noted in Fig. 2(a) as the horizontal direction, the angle between c-axis of Grain X1 and the ED is measured to be $\sim 55^\circ$, namely $\phi \sim 55^\circ$. This suggests that Grain X1 belongs to the primary texture component. Detailed TBDF TEM imaging was carried out near $[2\bar{1}\bar{1}0]$ zone axis under $\mathbf{g}=[01\bar{1}0]$ (Fig. 2(b)), and $\mathbf{g}=[0002]$ (Fig. 2(c)), respectively. Limited dislocations are present under $\mathbf{g}=[01\bar{1}0]$ (Fig. 2(b)) whereas no dislocation is visible under $\mathbf{g}=[0002]$ (Fig. 2(c)). Therefore these dislocations are all $\langle a \rangle$ type. In addition, the dislocation lines are mostly parallel to the trace of the basal plane, so the observed dislocations are mostly basal $\langle a \rangle$ dislocations. In fact, similar dislocation configurations have been observed in other grains (not shown here) with ϕ values between $45^\circ \sim 75^\circ$, which indicates that grains from the primary texture component are mostly recrystallized, having limited amounts of (basal) $\langle a \rangle$ dislocations. It is noted that occasionally stacking faults on basal plane were also observed in X-type grains, as the alloying element Y reduces the stacking fault energy of Mg [20, 26, 27].

Figure 3 presents another grain (Grain Y1 hereafter) near the $[2\bar{1}\bar{1}0]$ zone axis with minimum tilting. As this TEM specimen foil was sectioned normal to the ED and the ED is noted in Figure 3 as pointing into the viewing plane, the angle between c-axis of Grain Y1 and the ED is $\sim 90^\circ$, namely $\phi \sim 90^\circ$. This suggests that Grain Y1 belongs to the secondary texture component (i.e., extrusion basal texture). Similar to Grain X1, no dislocation is visible under $\mathbf{g}=[0002]$ with TBDF (Fig. 3(a)), whereas a fair amount of dislocations are visible under $\mathbf{g}=[0\bar{1}11]$ with TBDF (Fig. 3(b)) and WBDF (Fig. 3(c)), respectively. Therefore these dislocations are also $\langle a \rangle$ type. Compared to that in Grain X1, in Grain Y1: (1). There is higher density of dislocations; (2). While most dislocations are basal $\langle a \rangle$ type as they are parallel to the trace of basal plane, there are a few non-basal $\langle a \rangle$ dislocations as well; (3). Some subtle fringes parallel to the trace of basal plane can also be discerned, especially under WBDF (as highlighted by the white arrows in Fig. 3(b) and Fig. 3(c)). These fringes are caused by slightly inclined basal I2 stacking faults, which have been reported in detail by the same authors [26]. The density of I2 stacking faults (namely collinearly dissociated basal $\langle a \rangle$ dislocations) varies depending on how much each grain was deformed during hot extrusion [26].

Therefore, Grain X1 most likely represents recrystallized grains having few basal dislocations from the primary texture component, whereas Grain Y1 represents most likely deformed grains having a fairly high density of basal $\langle a \rangle$ dislocations from the secondary texture component. It is

also noted that there is a very limited amount of precipitates in the AE sample, as suggested by intensive TEM study (e.g., see the infrequent presence of precipitates in Figure 2 and Figure 3).

Figure 4 shows a representative APT reconstruction volume of the AE sample. Based on a total of five APT reconstruction volumes, it is concluded that: (1). no precipitates are present in any of the five reconstruction volumes due to the fact that there is a very limited number density and volume fraction of precipitates, and the majority of Y atoms are in Mg matrix solid solution; (2). Y solute atoms are generally randomly distributed in the Mg matrix solid solution (short range ordering of Y atoms, i.e., Y clustering, is observed[40]; however, this ordering or clustering is very weak as indicated by cluster analysis and partial radial distribution function that are not shown here); (3). the average concentration of Y in the Mg matrix is measured to be 1.59 ± 0.10 at.%, which is lower than the equilibrium solubility (~ 2.5 at.%) at the processing temperature of 350 °C, but notably higher than the equilibrium solubility (~ 0.6 at.%) at room temperature[28]. Hence, the material maintains a significant degree of supersaturated solid solution at room temperature, which is a result of the non-equilibrium thermo-mechanical processing. It is noted that with the lack of precipitates observed in TEM and APT, it is likely that the remaining Y atoms (~ 0.9 at.%) mostly distribute at grain boundaries, as grain boundary segregation of RE atoms has often been reported for Mg-RE alloys [41, 42]. The weak Y ordering or clustering is unlikely to significantly affect mechanical behavior, whereas the Y segregation at grain boundaries may have an effect on mechanical behavior.

3.2 Mechanical behavior and Texture evolution

Figure 5 shows the true stress-strain curves for both tensile and compression tests. Interestingly, there is a slight “yield drop” for both of the two curves (the enlarged inset), resulting in an upper yield point (UYP) and a lower yield point (LYP) for both tension and compression. The yield drop is most likely due to solid solution atoms impeding the initial movement of dislocations [43]. Overall the two curves are well aligned with each other, indicating dramatically reduced tension/compression asymmetry and strength differential [8]. The mechanical properties are summarized in the inset table. It is noted that unlike common Mg alloys in which the CYS is often much lower than the TYS [1, 44], in FG Mg-2.5Y actually the CYS is slightly higher than the TYS (based on both UYP and LYP). Therefore, the value of CYS/TYS is 1.02 (based on both UYP and LYP), which notably represents one of the closest ratios to unity heretofore reported for

wrought Mg alloys [10, 13, 14, 16, 45]. In addition, the UTS (379 MPa) is also very close to the UCS (403 MPa), namely $UCS/UTS = 1.06$.

In order to investigate the texture evolution around yielding, FG Mg-2.5Y samples tensile/compression tested to $\sim 2\%$ strain (namely immediately after the yield drop) were characterized by EBSD. Figure 6 presents the texture evolution from the AE sample to: compression tested to $\sim 2\%$ true strain (Comp-2% sample, Fig. 6(b)); and tensile tested to $\sim 2\%$ true strain (Tens-2% sample, Fig. 6(c)), respectively. Note that all IPFs presented here are with respect to the extrusion direction (ED), which is also the loading axis for both tension and compression. The texture of the AE sample is shown again in Fig. 6(a) (see also in Fig. 1(b)) to facilitate comparison. It is noted that after slight deformation, i.e., tension/compression by $\sim 2\%$, the texture change is very subtle, so the texture in the Comp-2% sample is still similar to that in the Tens-2% sample. It is also acknowledged that the maximum MUD value for the AE sample, Comp-2% sample or Tens-2% sample, highly depends on how many un-recrystallized large grains (e.g., large red grains in Figure 1 belonging to the extrusion basal texture component) are captured in each EBSD scan. Even though multiple scans of large areas for each of the three samples were performed, the population of these large grains still varies significantly in each EBSD map.

3.3 Dislocations and twins in compression tested FG Mg-2.5Y

In order to understand the unusual yield symmetry (see Section 3.2), as well as the difference in texture evolution between tension and compression, detailed EBSD and TEM studies focused on the Comp-2% sample were carried out. It is noted that the thin foil for TEM specimens was sectioned in the way that the thin foil/viewing plane was normal to the ED.

Figure 7 shows a representative set of EBSD data for the Comp-2% sample. Similar to that in Figure 1, the grains are colored based on the ϕ angle values. Therefore, if a grain, especially a red grain from the extrusion basal texture (i.e., secondary texture component) [1, 4], deforms by $\{10\bar{1}2\}$ twinning, blue colored substructure(s) should appear within a red grain due to the lattice re-orientation of $\sim 86^\circ$ caused by $\{10\bar{1}2\}$ twinning [46]. Based on these qualitative color codes, it is clear in Figure 7 that: (1). The majority of grains are green/yellow/orange due to the primary texture component; (2). There are still plenty of red grains due to the secondary texture

component; (3). Among thousands of grains, especially the red ones that would theoretically favor $\{10\bar{1}2\}$ twinning [4], only several grains (two of which are highlighted by black dashed circles) contain $\{10\bar{1}2\}$ twins in Figure 7. These results are consistent with the weak texture intensity of the $[0001]$ pole for the Comp-2% sample in Fig. 6(b). It is acknowledged that due to the limitation of resolution in EBSD, it is possible that some nano-sized $\{10\bar{1}2\}$ twins may actually exist in the Comp-2% sample but not detected by EBSD [27, 47]. Therefore, rigorous TEM study on Comp-2% sample was performed to characterize the defect structure in grains with various orientations.

Figure 8 shows the dislocation structure in the Comp-2% sample. In Fig. 8(a), after minimal tilting, one grain (Grain X2 hereafter) is oriented close to the $[7\bar{2}\bar{5}3]$ zone axis ($\mathbf{B} \sim [7\bar{2}\bar{5}3]$) and satisfying $\mathbf{g} = 0\bar{1}11$ (see inserted diffraction pattern). Therefore the angle between the c-axis of Grain X2 and the ED is $\sim 60^\circ$, namely $\phi \sim 60^\circ$, indicating that Grain X2 belongs to the primary texture component, similar to Grain X1 in the AE sample (in Figure 2). Straight lines of dislocation contrast can be clearly discerned, approximately aligned with the trace of the basal plane (highlighted by the white dashed line). In addition, the contrast of fringes aligned with the trace of the basal plane is caused by an inclined basal stacking fault [33, 48]. In Fig. 8(b), through significant tilting effort, Grain X2 is oriented near the $[2\bar{1}\bar{1}0]$ zone axis satisfying $\mathbf{g} = 0002$ (see inserted diffraction pattern). Compared to Fig. 8(a), no clear dislocation contrast could be discerned in Fig. 8(b), namely no $\langle c \rangle$ or $\langle c+a \rangle$ dislocations are present in Grain X2. Therefore, the dislocations observed in Grain X2 are all basal $\langle a \rangle$ dislocations, whereas the observed basal stacking fault is most likely I2 type even though the related partial dislocations are difficult to discern due to their weaker contrast [26].

Figure 9 presents another grain (Grain Y2) oriented near $[2\bar{1}\bar{1}0]$ zone axis after minimal tilting (Fig. 9(a)), and satisfying $\mathbf{g} = 01\bar{1}1$ (Fig. 9(b)). Therefore Grain Y2 is most likely from the extrusion texture, similar to Grain Y1 (in Figure 3). As the white dashed line again highlights the trace of basal plane, it is noted that some of the dislocations observed in Fig. 9(b) (as well as in Fig. 9(a)) are aligned with the basal plane, whereas some are not. Through additional minimal tilting, Grain B' also satisfies $\mathbf{g} = 0002$ near $[2\bar{1}\bar{1}0]$ zone axis in Fig. 9(c) (see inserted diffraction pattern). Similar to Grain X2, no $\langle c \rangle$ or $\langle c+a \rangle$ dislocations are present in Grain Y2. Therefore, the dislocations observed in Grain Y2 are all $\langle a \rangle$ dislocations, with some being basal

$\langle a \rangle$ dislocations, other being non-basal $\langle a \rangle$ dislocations (most likely being prismatic $\langle a \rangle$ dislocations).

Similar to Grain X2 and Grain Y2, plenty of basal/non-basal $\langle a \rangle$ dislocations have also been documented in many other grains with various orientations (not shown here), together with some basal stacking faults and very few $\langle c \rangle$ or $\langle c+a \rangle$ dislocations. Therefore, compared to the dislocation configuration in the AE sample (e.g., Figure 2 and 3), it is inferred that during the early stage (e.g., Stage I in Figure 5) of compressive deformation: (1). A majority of grains (i.e., X-type grains such as Grain X1 and X2) are deformed by basal slip, since the primary texture component favors basal slip; (2). Grains from the secondary texture component (i.e., Y-type grains such as Grain Y1 and Y2) are deformed primarily by prismatic slip; (3). $\{10\bar{1}2\}$ twinning activity is very limited even though the orientation of Y-type grains would favor twinning (see lack of twins in Figure 7 and absence of twins in Figure 9). Furthermore, due to the symmetry of dislocation slip, i.e., dislocations could move in one direction or the opposite direction depending on the stress state, $\langle a \rangle$ dislocation activity should also dominate the early stage of tensile deformation.

4 Discussion

4.1 Significantly reduced tension/compression yield asymmetry and strength differential in extruded FG Mg-2.5Y

Important trade-offs exist when using conventional approaches to reduce the plastic asymmetry of Mg or Mg alloys. As mentioned earlier, with the randomizing texture approach, strengthening is essentially very limited since the strengthening effect of reducing easy basal slip in basal textured Mg alloys is sacrificed [10]. In contrast, for the precipitation approach, since the precipitates generally impede twinning and/or slip, the yield strengths are commonly increased while ductility, especially tensile ductility, is compromised [16].

Therefore, the significantly reduced yield asymmetry (characterized by $CYS/TYS = 1.02$) and tension/compression strength differential (e.g., true stress-strain curves for tension and compression align close to each other in Figure 5) reported here for the extruded FG Mg-2.5Y are noteworthy. In fact, these results represent a new record in reducing yield asymmetry compared to published results, as well as reducing overall tension/compression asymmetry [3, 13,

14, 16, 18, 44, 49, 50]. For example, the value of UCS/UTS for FG Mg-2.5Y is 1.06, while in ref. [10] the values range between 1.29 and 1.60. However, from a scientific perspective, the underlying deformation mechanisms in this FG Mg-2.5Y, especially those responsible for the higher-than-usual CYS, are even more intriguing.

4.2 Microscopic lattice rotation/re-orientation mechanisms due to basal slip and $\{10\bar{1}2\}$ twinning

Based on the evident evolution of dislocation configurations in grains from the primary texture component, namely X-type grains with few basal $\langle a \rangle$ dislocations in the AE sample (Grain X1 in Figure 2) to a fair amount of basal $\langle a \rangle$ dislocations in the Comp-2% sample (Grain X2 in Figure 8), it is inferred that basal slip should play an important role in yielding and subsequent plastic deformation. Therefore, it is beneficial to examine in the following discussion how basal and non-basal slip, as well as the limited $\{10\bar{1}2\}$ twinning documented, would lead to texture evolution during deformation for FG Mg-2.5Y [51, 52].

Figure 10 schematically illustrates the microscopic difference in lattice rotation during tension and compression. Fig. 10(a) is a representative X-type grain (Grain X hereafter) from the primary texture component in the as-extruded FG Mg-2.5Y (e.g., Grain X1 in Figure 2). The angle between the c-axis (black dashed arrow) and the ED/deformation axis (vertical black solid arrow) is designated as φ_0 , while φ_0 is most probably between 45° and 75° . Therefore, basal slip would be favored in Grain X during tension or compression along the ED. The direction $\langle 11\bar{2}0 \rangle$ for basal slip is also highlighted, being perpendicular to the c-axis. In Fig. 10(b), during tensile deformation along the ED, Grain X is deformed by slip of basal $\langle a \rangle$ dislocations in one direction (e.g., $+1/3\langle 11\bar{2}0 \rangle$ direction). As a result of the incremental deformation by basal slip, the shape of Grain X also changes, namely becomes elongated along the ED. However, since Grain X is constrained by the surrounding grains in the polycrystal, it is forced to rotate counter-clockwise (CCW hereafter) to fit itself as much as possible into its surroundings. Consequently in Fig 10(c), the c-axis rotates CCW by $\Delta\theta$, leading to a larger angle φ_T (i.e., $\varphi_0 + \Delta\theta$) between c-axis and the ED as compared with the original angle φ_0 in Fig. 10(a). It is also noted that the slip direction $\langle 11\bar{2}0 \rangle$ rotates toward the ED. In contrast, during compressive along the ED (Fig. 10(d)), Grain X would also be deformed by basal $\langle a \rangle$ slip, but in an opposite direction (e.g., -

$1/3\langle 11\bar{2}0\rangle$ direction) compared to that in tension. Therefore, Grain X is forced to rotate clockwise (CW hereafter) to fit itself to its surroundings. As a result, the c-axis rotates CW by $\Delta\theta$ (Fig. 10(e)), leading to a smaller angle φ_c (i.e., $\varphi_0 - \Delta\theta$) between c-axis and the ED. At the same time, the slip direction $\langle 11\bar{2}0\rangle$ rotates away from the ED.

Fig. 10(f) is onerepresentative Y-type grain (Grain Y hereafter) from the secondary texture component, and its c-axis is nearly normal to the ED in the as-extruded FG Mg-2.5Y (e.g., red grains in Figure 7). Namely the angle between the c-axis and the ED (φ_0) is large, e.g., between roughly 75° and 90° . Therefore, $\{10\bar{1}2\}$ twinning deformation would theoretically be favored during compression along the ED. The $\langle 1\bar{1}00\rangle$ direction is also highlighted, since it is also involved in the re-orientation process of $\{10\bar{1}2\}$ twinning. In Fig. 10(g), during compression along the ED, $\{10\bar{1}2\}$ twinning may take place in Grain Y, leading to crystallographic re-orientation of $\sim 86^\circ$. Consequently, the angle between the c-axis and the ED (φ_c) becomes small, e.g., between 0° and 15° . In addition, the angle between the $\langle 1\bar{1}00\rangle$ direction and the ED increases. For example, the two red grains in Comp-2% sample (highlighted by black dashed circles in Figure 7) are deformed by $\{10\bar{1}2\}$ twinning, and thus the c-axis of the twinned region (the blue regions within the red grains) moves towards the ED. Nevertheless, even though $\{10\bar{1}2\}$ twinning is theoretically favored in Grain Y, in reality twinning rarely occurs, according to EBSD mapping (Figure 7) and TEM observation (Figure 9) in Comp-2% sample. Instead, non-basal $\langle a\rangle$ slip, most likely prismatic slip, is activated (Figure 7). The possible reasons are: (1). The presence of Y in Mg enhances prismatic slip, as has been previously reported [24, 53]; (2). The reduced grain size in FG Mg-2.5Y impedes twinning activity [54, 55].

It should be noted that during either tensile or compressive deformation along the ED, for grains deformed by basal slip, the lattice rotation occurs in a gradual manner and is proportional to the amount of deformation. In contrast, for grains like Grain Y during compressive deformation along the ED, grain re-orientation caused by $\{10\bar{1}2\}$ twinning happens in a more dramatic manner, namely small amounts of applied deformation leads to significant re-orientation [1]. For example, in-situ tensile deformation along the c-axis of the Mg single crystal pillars revealed that when a pillar was deformed to just 2~5% plastic strain, $\{10\bar{1}2\}$ twinning already consumed $\sim 50\%$ volume of the pillar, namely half of the lattice is re-oriented by $\sim 86^\circ$ [56]. Moreover, it is also acknowledged that polycrystalline deformation is not totally

analogous to single crystal deformation (i.e., Schmid's law). Particularly, the stress state of each grain is not necessarily equal to the macroscopic stress state in that local stress concentration commonly occurs for some grains [57-59]. Consequently, twinning/slip may still take place in grains with orientations that do not evidently favor twinning/slip. For example, even though the Schmid factors for basal slip in Grain Y2 in the Comp-2% sample (Figure 9) is very low since the basal plane is nearly parallel to the deformation axis, basal slip probably still took place.

4.3 Macroscopic texture evolution and deformation mechanisms during compressive/tensile deformation

Similar to what has been mentioned for Mg single crystal/grain deformation and lattice rotation/re-orientation in Section 4.2, during either tensile or compressive deformation for polycrystalline Mg, the texture evolution happens in a gradual manner for deformation by basal or non-basal slip. For example, Mayama et al. [8] numerically investigated the lattice rotation caused merely by basal slip in polycrystalline pure Mg with rolling texture during tension and compression. Prior to deformation, the average angle (κ hereafter) between the c-axis of the grains and the rolling direction (RD) was 79.73° ($\kappa_0=79.73^\circ$) with standard deviation (Δ hereafter) of 10.49° ($\Delta_0=10.49^\circ$). After 1% tensile deformation along the RD, $\kappa_T=80.24^\circ$ with $\Delta_T=10.19^\circ$. In other words, κ increased by 0.51° whereas Δ decreased by 0.30° . Conversely, after 1% compressive deformation along the RD, $\kappa_C=78.83^\circ$ with $\Delta_C=10.66^\circ$. This means that κ decreased by 0.90° whereas Δ increased by 0.17° . Similarly, during the Stage I compressive/tensile deformation (see Figure 5) in FG Mg-2.5Y, the average angle between the c-axis of the grains and the ED, namely ϕ herein, for the primary texture component would decrease/increase by probably less than 2° .

In contrast, for the secondary texture component in the AE sample during compressive deformation along the ED, texture evolution (if any) caused by $\{10\bar{1}2\}$ twinning should happen in an abrupt manner [1]. For example, in a typical Mg alloy with strong extrusion texture under compressive deformation along the ED, as little as ~2% macroscopic strain would lead to a $\{10\bar{1}2\}$ twin volume fraction of ~35% [60]. However in the FG Mg-2.5Y studied herein, there is very limited twinning activity (see Figure 7). The VPSC simulation results also confirm that the critical stress for prismatic slip is only ~60% of that for twinning. Therefore, the texture evolution is dominated by basal slip during early stage of deformation.

Interestingly, if basal slip with high values of Schmid factors (e.g., 0.43~0.50) dominates yield deformation for both compression and tension, one would expect the yield strengths (both CYS and TYS) to be very low, similar to the yield deformation in cast Mg alloys with basically no (or a random) texture[19]. However, the following unique effects of alloying Mg with Y solute atoms enabled extruded FG Mg-2.5Y to achieve both high TYS and CYS:

1. Excellent balance between solubility and solid solution strengthening for basal slip[25], especially supersaturated solid solution (~1.6 at.% Y based on APT data) in FG Mg-2.5Y produced through non-equilibrium processing. In fact, according to Yasi et al.[25], under ideal conditions, the CRSS for basal slip was calculated to increase by ~100 MPa with ~1.6 at.% Y in solid solution, i.e., $\Delta\text{CRSS}=100\text{ MPa}$.
2. Significantly reduced extrusion basal texture, and introduction of a rare-earth like texture, which favored basal slip upon yielding (average Schmid factor for basal slip ~0.3). Therefore, the solid solution strengthening alone contributed to both CYS and TYS. It also explains the “yield drop” for both tension and compression. As basal dislocation lines were initially pinned by Y atoms, slightly higher stress was needed to “un-pin” the dislocations[43].
3. Enhanced prismatic slip due to the presence of Y solute atoms[24], with the CRSS probably still larger than that for basal slip under both compression and tension.
4. Last but not least, the fine grain size (~1 μm) also likely played an important role in Hall-Petch strengthening[61], as well as suppressing $\{10\bar{1}2\}$ twinning activity[54], since in common basal textured Mg alloys, the easy $\{10\bar{1}2\}$ twinning often leads to low CYS [55].

4.4 Fundamental issues concerning $\langle c+a \rangle$ dislocations and basal stacking faults

Finally, it is informative to briefly discuss some fundamental issues concerning basal stacking faults and $\langle c+a \rangle$ dislocations, based on what was documented and/or what was absent in the FG Mg-2.5Y studied herein. Despite reportedly enhanced $\langle c+a \rangle$ dislocation activity in Mg-Y alloys [20, 22], in the FG Mg-2.5Y studied herein, few $\langle c+a \rangle$ dislocations were documented in either the as-processed sample (e.g., Figure 2 and 3 for the AE sample) or room temperature (RT) deformed samples (e.g., Figure 7 and 8 for the Comp-2% sample). In addition, a preliminary TEM study on Tens-2% sample (not shown here) indicates that $\langle c+a \rangle$ dislocations were unlikely to be active during tensile deformation either.

Pyramidal $\langle c+a \rangle$ slip is probably active during thermo-mechanical processing with the presence of high and multiaxial stress. So depending on the specific processing and cooling procedures, in the as-processed samples prior to RT deformation, there might be some “residual” $\langle c+a \rangle$ dislocations, or $\langle c \rangle$ dislocations[26] due to possible $\langle c+a \rangle \rightarrow \langle c \rangle + \langle a \rangle$ reactions[62] at RT. Therefore caution should be taken when relating RT mechanical behavior to $\langle c+a \rangle$ dislocations. Namely observation of $\langle c+a \rangle$ dislocations in RT deformed Mg-RE alloys does not necessarily mean that $\langle c+a \rangle$ slip is activated and operative during RT deformation, unless detailed comparison for dislocation configurations is made between the as-processed sample(s) and RT-deformed sample(s). For example, in the FG Mg-2.5Y studied herein, $\langle c+a \rangle$ slip did not play a significant role in deformation or texture evolution as revealed by both TEM and VPSC model.

In addition, basal stacking faults have also been observed in Mg-RE (e.g., Mg-Y) alloys [37]. However, the formation mechanisms for basal stacking faults have not been elucidated until recently [26], let alone the possible interaction(s) between stacking faults and slip/twinning. Moreover, in the FG Mg-2.5Y studied herein, most grains just contained a few stacking faults (e.g., Figure 8). Therefore, the possible strengthening effect (if any) of stacking faults in FG Mg-2.5Y should be very limited.

It is also acknowledged that what happened to the stacking faults in the AE sample during compression/tension was not clear. On the one hand, Figure 8 for the Comp-2% sample suggests that: (1). New $\langle a \rangle$ dislocations were activated parallel to the stacking fault(s) so that stacking faults may remain intact, e.g., note the presence of one stacking fault in Fig. 8(a); (2). Stacking faults might be destroyed by the possible glide and “re-association” of Shockley partials under compressive stress, resulting in the possibly reduced density of stacking faults in the Comp-2% sample, e.g., note the absence of stacking faults in Figure 9. Moreover, in an Mg-Ce alloy with reduced SFE deformed in-situ at ambient temperature, slip does not result in the formation of stacking faults[63, 64]. On the other hand, a preliminary TEM study on the Tens-2% sample suggests that the density of stacking faults might have increased after tensile deformation.

Therefore, stacking faults probably behave differently under tension and compression in FG Mg-2.5Y. Furthermore, this difference might be related to the subtle yet existing strength differential upon yielding, namely that the CYS was slightly higher than TYS (Figure 5). Further study is ongoing to clarify these issues.

5 Ongoing in-situ TEM study

As mentioned above, alloying with Y, a modified texture, and an FG microstructure are all factors that contributed to the observed yield symmetry and reduced strength differential. However, it is difficult to quantify the contribution from each factor, especially the latter two factors, as changing one factor would most likely change the other. Particularly in Mg-rare earth alloys, annealing as-processed alloys will result in a “rare-earth texture” [38]. In fact, the authors attempted to anneal the AE sample at 450 °C in order to obtain a “control group” of larger grain size. However, while the grain size indeed increased to ~2.7 μm , the texture also changed. The content of solid solution Y atoms was probably changed as well by annealing. In addition, compressive deformation was also performed on the consolidated Mg-2.5Y alloy (as-HIPed Mg-2.5Y [26, 27]) prior to extrusion. The microstructure of the as-HIPed sample (average grain size ~2.5 μm) and the compressed to ~2% strain sample was characterized by EBSD. Although as-HIPed Mg-2.5Y has random texture whereas as-extruded FG Mg-2.5Y has weakened basal texture, the distributions of ϕ angle are actually quite similar in both materials. Similar to the compressed FG Mg-2.5Y (Figure 7), there is a lack of twins in deformed HIPed Mg-2.5Y. It is noted that even many large red grains which would favor twinning during deformation do not contain twins. Nevertheless, it remains a challenging task to “decouple” the influence of extrinsic texture, grain size from intrinsic alloying effect on deformation mechanisms, unless more robust deformation tests and crystal plasticity modeling are performed.

Therefore, in-situ TEM deformation study was conducted on single-crystal, nano-sized pillars with various orientations prepared from AE sample using focused ion beam (FIB). Compared to recent reports on deforming pure Mg single-crystal nano-pillars [65], Mg-Y nano-pillars indeed showed significantly different deformation behavior. For example, the “lack of twinning” phenomenon for compressive deformation normal to the c-axis in bulk FG Mg-2.5Y was confirmed in multiple nano-pillars deformed in the same direction. Moreover, the CRSS values for basal slip, prismatic slip and even $\langle c+a \rangle$ slip were quantified respectively based on repeatable and consistent in-situ deformation data, and they are consistent with the dislocation configurations documented in deformed bulk FG Mg-2.5Y. Details will be reported in a follow-up study.

6 Conclusions

Here we report novel results for an extruded FG Mg-2.5Y alloy that exhibits virtual tension/compression yield symmetry when deformed in the extrusion direction. Moreover, the strength differential between compression and tension was significantly reduced as well. The key findings and conclusions are as follows:

1. For both compression and tension, the modified primary “rare-earth” like texture favored basal slip, whereas the secondary extrusion texture favored prismatic slip. Moreover, the fine grain size likely suppressed $\{10\bar{1}2\}$ twinning activity during compression. Therefore, the dominant deformation modes upon yielding for both compression and tension were basal slip and prismatic slip, resulting in a virtual yield symmetry.
2. The significant strengthening effect of supersaturated Y solid solution ensured that decent strength was maintained for both compression and tension, while the strength differential was reduced at the same time. Moreover, the presence of Y also enhanced prismatic slip.
3. The activity of $\langle c+a \rangle$ dislocations and basal stacking faults was also discussed. While $\langle c+a \rangle$ dislocations were unlikely to be active, the possibly different behavior of stacking faults under compression and tension was unclear and deserves further study.

Acknowledgements Dr. T. Topping’s assistance with mechanical tests and Dr. T. Hu’s and Dr. L. Jiang’s discussion on TEM are highly appreciated. This work was supported by National Science Foundation (NSF CMMI-1437327) and 111 Project of China (No. B13035). Prof. David N. Seidman and Dr. Dieter Isheim are acknowledged for providing access to atom probe and other facilities at Northwestern University Center for Atom Probe Tomography (NUCAPT).

References

- [1] Wang YN, Huang JC. The role of twinning and untwinning in yielding behavior in hot-extruded Mg–Al–Zn alloy. *Acta Mater* 2007;55:897-905.
- [2] Barnett MR, Sullivan A, Stanford N, Ross N, Beer A. Texture selection mechanisms in uniaxially extruded magnesium alloys. *Scr Mater* 2010;63:721-4.
- [3] Lee M-G, Wagoner RH, Lee JK, Chung K, Kim HY. Constitutive modeling for anisotropic/asymmetric hardening behavior of magnesium alloy sheets. *Int J Plast* 2008;24:545-82.
- [4] Barnett MR. Twinning and the ductility of magnesium alloys: Part I: “Tension” twins. *Materials Science and Engineering: A* 2007;464:1-7.
- [5] Zhang D, Jiang L, Zheng B, Schoenung JM, Mahajan S, Lavernia EJ, et al. Deformation Twinning (Update). *Reference Module in Materials Science and Materials Engineering*: Elsevier; 2016.

- [6] Bohlen J, Nürnberg MR, Senn JW, Letzig D, Agnew SR. The texture and anisotropy of magnesium–zinc–rare earth alloy sheets. *Acta Mater* 2007;55:2101-12.
- [7] Christian JW, Mahajan S. DEFORMATION TWINNING. *Prog Mater Sci* 1995;39:1-157.
- [8] Mayama T, Aizawa K, Tadano Y, Kuroda M. Influence of twinning deformation and lattice rotation on strength differential effect in polycrystalline pure magnesium with rolling texture. *Computational Materials Science* 2009;47:448-55.
- [9] Hirsch J, Al-Samman T. Superior light metals by texture engineering: Optimized aluminum and magnesium alloys for automotive applications. *Acta Mater* 2013;61:818-43.
- [10] Bohlen J, Yi S, Letzig D, Kainer KU. Effect of rare earth elements on the microstructure and texture development in magnesium–manganese alloys during extrusion. *Materials Science and Engineering: A* 2010;527:7092-8.
- [11] Beyerlein IJ, Toth LS. Texture evolution in equal-channel angular extrusion. *Prog Mater Sci* 2009;54:427-510.
- [12] Kim K-H, Suh B-C, Bae JH, Shim M-S, Kim S, Kim NJ. Microstructure and texture evolution of Mg alloys during twin-roll casting and subsequent hot rolling. *Scr Mater* 2010;63:716-20.
- [13] Singh A, Osawa Y, Somekawa H, Mukai T. Ultra-fine grain size and isotropic very high strength by direct extrusion of chill-cast Mg–Zn–Y alloys containing quasicrystal phase. *Scr Mater* 2011;64:661-4.
- [14] Singh A, Somekawa H, Mukai T. Compressive strength and yield asymmetry in extruded Mg–Zn–Ho alloys containing quasicrystal phase. *Scr Mater* 2007;56:935-8.
- [15] Al-Samman T, Li X. Sheet texture modification in magnesium-based alloys by selective rare earth alloying. *Materials Science and Engineering: A* 2011;528:3809-22.
- [16] Stanford N, Geng J, Chun YB, Davies CHJ, Nie JF, Barnett MR. Effect of plate-shaped particle distributions on the deformation behaviour of magnesium alloy AZ91 in tension and compression. *Acta Mater* 2012;60:218-28.
- [17] Basu I, Al-Samman T. Triggering rare earth texture modification in magnesium alloys by addition of zinc and zirconium. *Acta Mater* 2014;67:116-33.
- [18] Robson JD, Stanford N, Barnett MR. Effect of precipitate shape on slip and twinning in magnesium alloys. *Acta Mater* 2011;59:1945-56.
- [19] Jain J, Poole WJ, Sinclair CW, Gharghoury MA. Reducing the tension–compression yield asymmetry in a Mg–8Al–0.5Zn alloy via precipitation. *Scr Mater* 2010;62:301-4.
- [20] Sandlöbes S, Friák M, Zaefferer S, Dick A, Yi S, Letzig D, et al. The relation between ductility and stacking fault energies in Mg and Mg–Y alloys. *Acta Mater* 2012;60:3011-21.
- [21] Zhang D, Zheng B, Zhou Y, Mahajan S, Lavernia EJ. Prism stacking faults observed contiguous to a {10-12} twin in a Mg–Y alloy. *Scr Mater*.
- [22] Sandlöbes S, Zaefferer S, Schestakow I, Yi S, Gonzalez-Martinez R. On the role of non-basal deformation mechanisms for the ductility of Mg and Mg–Y alloys. *Acta Mater* 2011;59:429-39.
- [23] Cottam R, Robson J, Lorimer G, Davis B. Dynamic recrystallization of Mg and Mg–Y alloys: Crystallographic texture development. *Materials Science and Engineering: A* 2008;485:375-82.
- [24] Yasi JA, Hector Jr LG, Trinkle DR. Prediction of thermal cross-slip stress in magnesium alloys from a geometric interaction model. *Acta Mater* 2012;60:2350-8.
- [25] Yasi JA, Hector Jr LG, Trinkle DR. First-principles data for solid-solution strengthening of magnesium: From geometry and chemistry to properties. *Acta Mater* 2010;58:5704-13.

- [26] Zhang D, Jiang L, Schoenung JM, Mahajan S, Lavernia EJ. TEM study on relationship between stacking faults and non-basal dislocations in Mg. *Philos Mag* 2015;1-22.
- [27] Zhang D, Zheng B, Zhou Y, Mahajan S, Lavernia EJ. Prism stacking faults observed contiguous to a {10-12} twin in a Mg–Y alloy. *Scr Mater* 2014;76:61-4.
- [28] Guo C, Du Z, Li C. A thermodynamic description of the Mg–Pr–Y system. *Calphad* 2008;32:177-87.
- [29] Guo C, Du Z, Li C. A thermodynamic description of the Gd–Mg–Y system. *Calphad* 2007;31:75-88.
- [30] Lavernia EJ, Srivatsan TS. The rapid solidification processing of materials: science, principles, technology, advances, and applications. *J Mater Sci* 2010;45:287-325.
- [31] Ma K, Wen H, Hu T, Topping TD, Isheim D, Seidman DN, et al. Mechanical behavior and strengthening mechanisms in ultrafine grain precipitation-strengthened aluminum alloy. *Acta Mater* 2014;62:141-55.
- [32] Wen H, Topping TD, Isheim D, Seidman DN, Lavernia EJ. Strengthening mechanisms in a high-strength bulk nanostructured Cu–Zn–Al alloy processed via cryomilling and spark plasma sintering. *Acta Mater* 2013;61:2769-82.
- [33] Williams DB, Carter CB. Transmission electron microscopy: a textbook for materials science: Springer; 2009.
- [34] Vaidya S, Mahajan S. ACCOMMODATION AND FORMATION OF (1121) TWINS IN CO SINGLE-CRYSTALS. *Acta Metallurgica* 1980;28:1123-31.
- [35] Li B, Ma E, Ramesh KT. Dislocation configurations in an extruded ZK60 magnesium alloy. *Metall Mater Trans A-Phys Metall Mater Sci* 2008;39A:2607-14.
- [36] Agnew SR, Horton JA, Yoo MH. Transmission electron microscopy investigation of $\langle c+a \rangle$ dislocations in Mg and alpha-solid solution Mg–Li alloys. *Metall Mater Trans A-Phys Metall Mater Sci* 2002;33:851-8.
- [37] Sandlöbes S, Pei Z, Friák M, Zhu LF, Wang F, Zaefferer S, et al. Ductility improvement of Mg alloys by solid solution: Ab initio modeling, synthesis and mechanical properties. *Acta Mater* 2014;70:92-104.
- [38] Stanford N, Barnett MR. The origin of “rare earth” texture development in extruded Mg-based alloys and its effect on tensile ductility. *Materials Science and Engineering: A* 2008;496:399-408.
- [39] Al-Samman T, Gottstein G. Dynamic recrystallization during high temperature deformation of magnesium. *Materials Science and Engineering: A* 2008;490:411-20.
- [40] Nie JF, Wilson NC, Zhu YM, Xu Z. Solute clusters and GP zones in binary Mg–RE alloys. *Acta Mater* 2016;106:260-71.
- [41] Hadorn JP, Sasaki TT, Nakata T, Ohkubo T, Kamado S, Hono K. Solute clustering and grain boundary segregation in extruded dilute Mg–Gd alloys. *Scr Mater* 2014;93:28-31.
- [42] Bugnet M, Kula A, Niewczas M, Botton GA. Segregation and clustering of solutes at grain boundaries in Mg–rare earth solid solutions. *Acta Mater* 2014;79:66-73.
- [43] Brindley BJ, Worthington PJ. Yield-point phenomena in substitutional alloys. *International Materials Reviews* 1970;15:101-14.
- [44] Laser T, Hartig C, Nürnberg MR, Letzig D, Bormann R. The influence of calcium and cerium mischmetal on the microstructural evolution of Mg–3Al–1Zn during extrusion and resulting mechanical properties. *Acta Mater* 2008;56:2791-8.

- [45] Lentz M, Klaus M, Beyerlein IJ, Zecevic M, Reimers W, Knezevic M. In situ X-ray diffraction and crystal plasticity modeling of the deformation behavior of extruded Mg–Li–(Al) alloys: An uncommon tension–compression asymmetry. *Acta Mater* 2015;86:254-68.
- [46] Partridge PG. The crystallography and deformation modes of hexagonal close-packed metals. *Metallurgical reviews* 1967;12:169-94.
- [47] El Kadiri H, Kapil J, Oppedal AL, Hector Jr LG, Agnew SR, Cherkaoui M, et al. The effect of twin–twin interactions on the nucleation and propagation of twinning in magnesium. *Acta Mater* 2013;61:3549-63.
- [48] Zhu YM, Morton AJ, Weyland M, Nie JF. Characterization of planar features in Mg–Y–Zn alloys. *Acta Mater* 2010;58:464-75.
- [49] Bohlen J, Dobroň P, Swiostek J, Letzig D, Chmelík F, Lukáč P, et al. On the influence of the grain size and solute content on the AE response of magnesium alloys tested in tension and compression. *Materials Science and Engineering: A* 2007;462:302-6.
- [50] Hantzsche K, Bohlen J, Wendt J, Kainer KU, Yi SB, Letzig D. Effect of rare earth additions on microstructure and texture development of magnesium alloy sheets. *Scr Mater* 2010;63:725-30.
- [51] Lebensohn RA, Tomé CN. A self-consistent anisotropic approach for the simulation of plastic deformation and texture development of polycrystals: Application to zirconium alloys. *Acta Metallurgica et Materialia* 1993;41:2611-24.
- [52] Tomé CN. Lectures from Computational Materials Science Summer School at Texas A&M University 2014.
- [53] Suzuki M, Sato H, Maruyama K, Oikawa H. Creep behavior and deformation microstructures of Mg–Y alloys at 550 K. *Materials Science and Engineering: A* 1998;252:248-55.
- [54] Barnett MR, Keshavarz Z, Beer AG, Atwell D. Influence of grain size on the compressive deformation of wrought Mg–3Al–1Zn. *Acta Mater* 2004;52:5093-103.
- [55] Ghaderi A, Barnett MR. Sensitivity of deformation twinning to grain size in titanium and magnesium. *Acta Mater* 2011;59:7824-39.
- [56] Yu Q, Qi L, Chen K, Mishra RK, Li J, Minor AM. The Nanostructured Origin of Deformation Twinning. *Nano Letters* 2012;12:887-92.
- [57] Beyerlein IJ, McCabe RJ, Tomé CN. Effect of microstructure on the nucleation of deformation twins in polycrystalline high-purity magnesium: A multi-scale modeling study. *Journal of the Mechanics and Physics of Solids* 2011;59:988-1003.
- [58] Barnett MR. 3 - Twinning and its role in wrought magnesium alloys. In: Bettles C, Barnett M, editors. *Advances in Wrought Magnesium Alloys*: Woodhead Publishing; 2012. p. 105-43.
- [59] Niezgoda SR, Kanjarla AK, Beyerlein IJ, Tomé CN. Stochastic modeling of twin nucleation in polycrystals: An application in hexagonal close-packed metals. *Int J Plast* 2014;56:119-38.
- [60] Barnett MR, Nave MD, Ghaderi A. Yield point elongation due to twinning in a magnesium alloy. *Acta Mater* 2012;60:1433-43.
- [61] Meyers MA, Chawla KK. *Mechanical Behavior of Materials*. 2009.
- [62] Yoo MH, Agnew SR, Morris JR, Ho KM. Non-basal slip systems in HCP metals and alloys: source mechanisms. *Mater Sci Eng A-Struct Mater Prop Microstruct Process* 2001;319:87-92.
- [63] Zhang J, Dou Y, Liu G, Guo Z. First-principles study of stacking fault energies in Mg-based binary alloys. *Computational Materials Science* 2013;79:564-9.
- [64] Ye J, Mishra RK, Sachdev AK, Minor AM. In situ TEM compression testing of Mg and Mg–0.2 wt.% Ce single crystals. *Scr Mater* 2011;64:292-5.

[65] Yu Q, Qi L, Mishra RK, Li J, Minor AM. Reducing deformation anisotropy to achieve ultrahigh strength and ductility in Mg at the nanoscale. Proceedings of the National Academy of Sciences 2013;110:13289-93.

List of Figures and Tables:

Figure 1 Microstructure of as-extruded FG Mg-2.5Y (AE sample). (a). EBSD map, showing the average grain size is $\sim 1 \mu\text{m}$. The rainbow color bar represents different angle between c-axis and the ED (ϕ angle). (b). Inverse pole figure with respect to the ED, showing a weak extrusion basal texture. (c). The distribution of ϕ angle.

Figure 2 Grain X1 near $[2\bar{1}\bar{1}0]$ zone axis viewed in the thin foil parallel to the extrusion direction (ED) in AE sample. With minimum tilting, it is oriented in ways that satisfy two beam conditions. (a) and (b), Grain X1 at $[2\bar{1}\bar{1}0]$ zone axis ($\mathbf{B}=[2\bar{1}\bar{1}0]$); (c) and (d), $\mathbf{g}=[01\bar{1}0]$ in TBDF; (e) and (f), $\mathbf{g}=[0002]$ in TBDF.

Figure 3 Grain Y1 near $[2\bar{1}\bar{1}0]$ zone axis($\mathbf{B}=[2\bar{1}\bar{1}0]$) viewed in the thin foil normal to the ED in AE sample. With minimum tilting, it is oriented in ways that satisfy two beam or weak beam dark field conditions: (a) $\mathbf{g}=[0002]$ in TBDF; (b) and (c), $\mathbf{g}=[0\bar{1}11]$ in TBDF and WBDF, respectively.

Figure 4 A representative atom-probe tomographic 3D reconstruction of the AE sample, which contains 18 M atoms. Pink and blue dots represent Mg and Y atoms, respectively. 100% Y atoms are displayed, whereas only 1% Mg atoms are shown for clarity.

Figure 5 True stress-strain curves for tension and compression tests in the extrusion direction (ED). The enlarged inset figure shows the slight “yield drop” ($\sim 11 \text{ MPa}$) phenomena for both tension and compression. The inset table summarizes the mechanical properties. Note that the yield asymmetry (CYS/TYS) and ultimate strength asymmetry (UCS/UTS) approach to unity.

Figure 6 Texture evolution from (a) as-extruded Mg-2.5Y (AE sample) to: (b). compressed to $\sim 2\%$ true strain (Comp-2% sample), and (c). tensile tested to $\sim 2\%$ true strain (Tens-2% sample), respectively. All the inverse pole figures presented here are with respect to the extrusion direction (ED).

Figure 7 A representative set of EBSD data for Comp-2% sample. The rainbow color bar represents different angle between c-axis and the ED. Twograins containing $\{10\bar{1}2\}$ twins are highlighted by black dashed circles in the map.

Figure 8 Dislocation structure in Comp-2% sample. (a). Grain X2 near the $[7\bar{2}\bar{5}3]$ zone axis ($\mathbf{B}\sim[7\bar{2}\bar{5}3]$) while satisfying $\mathbf{g}=0\bar{1}11$ after minimal tilting. Dislocation contrast aligns with the trace of the basal plane (highlighted by the white dashed line). An inclined basal stacking fault (BSF) is also present; (b), after significant tilting, Grain X2 is oriented near the $[2\bar{1}\bar{1}0]$ zone axis ($\mathbf{B}\sim[2\bar{1}\bar{1}0]$) while satisfying $\mathbf{g}=0002$.

Figure 9 Dislocation structure in Comp-2% sample. (a), Grain Y2 at the $[2\bar{1}\bar{1}0]$ zone axis ($\mathbf{B}=[2\bar{1}\bar{1}0]$), dislocation contrast can already be seen;(b), $\mathbf{g}=01\bar{1}1$ under WBDF after minimal tilting; (c), $\mathbf{g}=0002$ under WBDF. The white dashed lines highlight the trace of the basal plane.

Figure 10 Schematic illustrations for microscopic lattice rotation mechanisms during deformation. (a). One representative grain (Grain X) from the primary texture component, with c-axis inclined with respect to the ED. Grain X would favor basal slip during either tensile or compression deformation along the ED. (b) and (c), Due to the physical confinement from the surrounding grains during tensile deformation along the ED, Grain X rotates in the way that the c-axis moves further away from the ED. (d) and (e), during compressive deformation along the ED, c-axis of Grain X moves towards the ED. (f) One representative grain (Grain Y) from the secondary texture component, with c-axis being nearly perpendicular to the ED. (g) During compressive deformation along the ED, Grain Y would deform by either $\{10\bar{1}2\}$ twinning or prismatic slip.

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10